

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

4197-112

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

10/070624

INTERNATIONAL APPLICATION NO.

PCT/DE00/03410

INTERNATIONAL FILING DATE

29 September 2000

PRIORITY DATE CLAIMED

6 October 1999

TITLE OF INVENTION

METHOD AND DEVICE FOR PRODUCING CELLULOSED SHAPED BODIES

APPLICANT(S) FOR DO/EO/US

NIEMZ, Frank-Gunter; MEYER, Wilhelm; MAINZER ALTHOF, Tanja

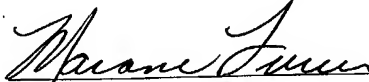
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)) ***(Unsigned)**
10. ☒ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5))

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment
14. ☐ A substitute specification
15. ☒ A small entity statement
16. ☐ Other items or information

NOTE: This application is being filed with an unsigned Oath or Declaration under the provisions of 37 CFR § 1.53 in order that applicants may secure a filing date of March 4, 2002. Upon receipt of a "Notice to File Missing Parts - Filing Date Granted," an executed Declaration and Power of Attorney, will be filed in the Patent and Trademark Office. The undersigned agent affirmatively states that she has been duly authorized and appointed to file this application on behalf of the applicants and that the Declaration and Power of Attorney to be filed hereafter will confirm the undersigned agent's authorization and appointment. Applicants are entitled to small entity status within the meaning of 37 CFR § 1.9.

17. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS		PTO USE ONLY	
Basic National Fee (37 CFR 1.492(a)(1)-(5)) Search Report has been prepared by the EPO or JPO \$860.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) \$0.00 No International preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$0.00 Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$1000.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) \$0.00				JC13 Rec'd PCT/PTO 04 MAR 2002			
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$ 860.00			
* Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e))				\$			
Claims	Number Filed	Number Extra	Rate				
Total Claims	13-20 =	0	X \$18.00	\$			
Independent Claims	2-3 =	0	X \$80.00	\$			
Multiple dependent claim(s) (if applicable)			+ \$270.00	\$			
TOTAL OF ABOVE CALCULATIONS =				860.00			
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28)				\$ 430.00			
SUBTOTAL =				\$ 430.00			
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 Months from the earliest claimed priority date (37 CFR 1.492(f)).				\$			
TOTAL NATIONAL FEE =				\$ 430.00			
Fee for recording the enclosed assignment (37 CFR 1.21(h)) The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property				\$			
TOTAL FEE ENCLOSED =				\$ 430.00			
				Amount to be:			
				refunded			
				Charged			
a. <input checked="" type="checkbox"/> A check in the amount of <u>\$430.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>08-3284</u> . A duplicate copy of this sheet is enclosed.							
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not yet been met, a petition to revive (37 CFR 1.127(a) or (b)) must be filed and granted to restore the application to pending status.							
SEND ALL CORRESPONDENCE TO: Steven J. Hultquist Intellectual Property/Technology Law P. O. Box 14329 Research Triangle Park, NC 27709				 MARIANNE FUIERER Registration No. 39,983			

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JC13 Rec'd PCT/PTO 0 4 MAR 2002

4197-112
PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

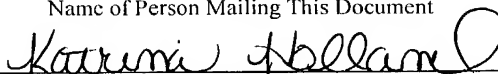
In re Application of: Frank-Gunter Niemz, et al.
Application No.: New U.S. National Stage Application of
PCT International Application No. PCT/DE00/03410
International Filing Date: 29 September 2000
Priority Date Claimed: 6 October 1999 (German Appl. No. 199 47 910.0)
15 October 1999 (German Appl. No. 199 49 726.5)
U.S. National Phase Filing Date: Date of mailing identified below
Title: **METHOD AND DEVICE FOR PRODUCING
CELLULOSED SHAPED BODIES**

EXPRESS MAIL CERTIFICATE

I hereby certify that I am mailing the attached documents to the
Commissioner for Patents on the date specified, in an envelope
addressed to the Commissioner for Patents, Box Patent
Application, Washington, DC 20231, and Express Mailed under
the provisions of 37 CFR 1.10.

Katrina Holland

Name of Person Mailing This Document



Signature

March 4, 2002

Date

EV037733168US

Express Mail Label Number

PRELIMINARY AMENDMENT

Commissioner for Patents
BOX PATENT APPLICATION
Washington, D.C. 20231

Sir:

Prior to examination of the above-identified new national phase patent application, please amend the application, as follows:

In the Specification¹

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03410 filed September 29, 2000, which in turn claims priority of German Patent Application Nos. 199 47 910.0 filed October 6, 1999 and 199 49 726.5 filed October 15, 1999.

In the Abstract

Please amend the abstract to read as follows:

The invention relates to a method for producing cellulosed shaped bodies. (a) Cellulose and an aqueous aminoxide are mixed, whereby a suspension is produced. (b) A cellulose solution is produced from the suspension, whereby water evaporates. (c) A first property and a second property of the produced cellulose solution is measured and the measured value is used for correcting the composition of the solution and (d) the cellulose solution is extruded through a gap of air and

¹ Applicants has provided a marked-up version of amended paragraph, abstract and claims 1-10 in Appendix A, and a clean set of all pending claims, amended to date, in Appendix B.

into an aqueous regenerating bath where said solution is coagulated to form shaped bodies.

In the Claims

Please amend claims 1-10 to read as follows:

1. A process for controlling the composition of a solution in the production of cellulosic shaped bodies, the process comprising:

- (a) mixing cellulose and a water-containing amine oxide to form a suspension,
- (b) creating a cellulose solution from the suspension by vaporization of water,
- (c) measuring a first property of the cellulose solution and a second property different from the first property wherein the measured values of the first and second properties are used to calculate the actual composition of the solution and their deviation from values of a target composition is used to adjust the composition of the solution; and
- (d) extruding the cellulose solution through an air gap into an aqueous regeneration bath where it is coagulated to form shaped bodies that are capable of undergoing further processing steps.

2. The process according to claim 1, wherein the first and second properties of the cellulose solution are selected from the group consisting of: relative permittivity, inductive conductivity, microwave absorption, refractive index, density, water content, and ultrasonic speed.

3. The process according to claim 2, further comprising measuring the cellulose solution's temperature, and the measured values of the first and second properties are adjusted on the basis of the measured temperature, wherein the timing of the temperature measuring is selected from the group consisting of during the measuring of the first and second property, shortly before the measuring of the first and second property and after measuring of the first and second property.
4. The process according to claim 2, wherein at least one of the first and second property is measured in-line.
5. The process according to claim 2, wherein the solution composition is adjusted by manual intervention in the dosing of the suspension components of step (a).
6. The process according to claim 2, wherein operating conditions are adjusted by manual intervention in the operating conditions of step (b).
7. A device for performing a process for controlling the composition of a cellulose solution comprising cellulose and a water-containing amine oxide used in the production of cellulosic shaped bodies the device comprising:
 - (a) a slurring apparatus communicatively connected with at least two metering devices for metering input of the cellulose and water-containing amine oxide;
 - (b) a dissolving and vaporization arrangement connected to the slurring apparatus;

(c) an extruding device connected by a conduit to the dissolving and vaporizing arrangement and having a regeneration bath attached downstream therefrom;

(d) at least two measuring instruments contacting the conduit for measuring a first and a second property of the cellulose solution passing through the conduit; and

(e) a controlling circuit for controlling the composition of the cellulose solution by determining the actual composition of the cellulose solution in the conduit from the measured values of the first and second property and to determine the deviation from a preset target composition and adjust metering devices to correct for any deviation.

8. The device according to claim 7, where in the first and second property of the cellulose solution are different and selected from the group consisting of: relative permittivity, inductive conductivity, microwave absorption, refractive index, density, water content, and ultrasonic speed.
9. The device according to claim 8, further comprising a temperature measuring instrument for measuring the temperature of the cellulose solution and to compensate for temperature in the measured values of measuring instruments.
10. The device according to claim 7, wherein the measuring instruments, a microprocessor and the metering devices form the controlling circuit.

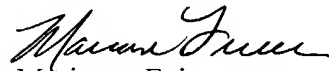
Please add the following claims.

11. The device according to claim 7, wherein the measuring instruments, a microprocessor and the dissolving and vaporizing arrangement form the controlling circuit.
12. The device according to claim 7, wherein the measuring instruments are arranged consecutively in the conduit.
13. The device according to claim 9, wherein the measuring temperature instrument is positioned between the measuring instrument for the first property and the measuring instrument for the second property.

REMARKS

It is requested that the examination and prosecution of this application proceed on the basis of these amended and new claims 1-13.

Respectfully submitted,



Marianne Fuierer

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APPENDIX A

In the Specification

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03410 filed September 29, 2000, which in turn claims priority of German Patent Application Nos. 199 47 910.0 filed October 6, 1999 and 199 49 726.5 filed October 15, 1999.

In the Abstract

Please amend the abstract to read as follows:

ABSTRACT

The invention relates to a method for producing cellulosed shaped bodies. (a) Cellulose and an aqueous aminoxide are mixed, whereby a suspension is produced. (b) A cellulose solution is produced from the suspension, whereby water evaporates. (c) A first property [or a physical behavior] and a second property of the produced cellulose solution is measured and the measured value is used for correcting the composition of the solution and (d) the cellulose solution is extruded through a gap of air and into an aqueous regenerating bath where said solution is coagulated to form shaped bodies. [which can go through additional treatment steps. The invention is characterized in that a second property of the

produced cellulose solution is measured in step (c), said second property being different from the first property. The real composition of the solution is calculated by means of the measured values of both properties. The deviation from a desired composition is used for controlling the composition of the solution. The inventive method enables to exactly control the composition of the spinning solution.]

In the Claims

Please amend claims 1-10 to read as follows:

1. A process for controlling the composition of a solution [or solutions] in the production of cellulosic shaped bodies, the process comprising: [in which]

- (a) mixing cellulose and a water-containing amine oxide [are mixed] to form a suspension,
- (b) creating a cellulose solution [is created] from the suspension by vaporization of water,
- (c) measuring a first property [or physical behavior] of the cellulose solution and a second property different from the first property wherein the measured values of the first and second properties are [formed thereby is measured and the measured value is] used to calculate the actual composition of the solution [is calculated on the basis of measured values for the two properties,] and their deviation from values of a target composition is used to adjust the composition of the solution; [used to adjust the composition of the solution,] and

(d) extruding the cellulose solution [is extruded] through an air gap into an aqueous regeneration bath where it is coagulated to form shaped bodies that are capable of undergoing further processing steps.,

characterized in that

in step (c), a second property of the cellulose solution, which is different from the first property, is measured,

the actual composition of the solution is calculated on the basis of measured values for the two properties, and their deviation from a target composition is used to adjust the composition of the solution.]

2. The [A] process according to claim 1, wherein [characterized in that] the first and second [two] properties of the cellulose [spinning solution] are selected from the group consisting of: [chosen from the] relative permittivity [permittivities], [the] inductive conductivity, [the] microwave absorption, [the] refractive index, [the] density, [the] water content, and [the] ultrasonic speed.
3. The [A] process according to claim [1 or] 2, further comprising measuring [characterized in that the temperature of] the cellulose solution's temperature [is measured during or shortly before or after the two properties are measured], and the measured values of the first and second [two] properties are adjusted on the basis of the measured temperature, wherein the timing of the temperature measuring is selected from the group consisting of during the measuring of the first and second property, shortly before the measuring of the first and second property and after measuring of the first and second property.

4. The [A] process according to claim 2, wherein [any of claims 1 to 3, characterized in that] at least one of the first and second property [two properties] is measured in-line.
5. The [A] process according to claim 2 wherein [any of claims 1 to 4, characterized in that] the solution composition is adjusted by manual intervention in the dosing of the suspension components of step (a).
6. The [A] process according to claim 2 wherein [any of claims 1 to 5, characterized in that the] operating conditions are adjusted by manual intervention in the operating conditions of step (b).
7. A device for performing a process for controlling the composition of a cellulose solution comprising cellulose and a water-containing amine oxide used in the production of cellulosic shaped bodies [the process according to any of claims 1 to 6, having] the device comprising:
 - (a) a slurring apparatus [(2)] communicatively connected [furnished] with at least two metering devices for metering input of the cellulose and water-containing amine oxide; [(1^a, 1^b),]
 - (b) a dissolving and vaporization arrangement [(3)] connected to the slurring apparatus; [(2) and]
 - (c) an extruding device [(4)] connected by a conduit [(8)] to the dissolving and vaporizing arrangement [(3)] and having a regeneration bath [(5)] attached downstream therefrom; [, and]

(d) [characterized in that] at least two measuring instruments [apparatus (9-11)] contacting the conduit for measuring a first and a second property of the cellulose solution passing through the conduit; and

(e) [that constitutes part of] at least one controlling circuit for controlling the composition of the cellulose solution [is disposed in the conduit (8) for the purpose of measuring two different properties of the cellulose solution] by determining the actual composition of the cellulose solution in the conduit from the measured values of the first and second property and to determine the deviation from a preset target composition and adjust metering devices to correct for any deviation.

8. The [A] device according to claim 7, where in the first and second property of the cellulose solution are different and selected from the group consisting of: relative permittivity, inductive conductivity, microwave absorption, refractive index, density, water content, and ultrasonic speed. [is selected characterized in that the measuring apparatus consists of two instruments (9, 10) for the purpose of measuring different properties of the solution].
9. The [A] device according to claim 8, further comprising [characterized in that the measuring apparatus includes an] a temperature measuring instrument [(11)] for [the purpose of] measuring the temperature of the cellulose solution and to compensate [compensating] for temperature in the measured values of measuring instruments [(9, 10)].
10. The [A] device according to claim [any of claims] 7 [to 9], wherein [characterized in that] the measuring instruments, [measurement apparatus (9-11) together with]

a microprocessor [(12)] and the metering devices [(1^a,
1^b) or the dissolving and vaporizing arrangement (3)]
forms] form the controlling circuit [a control circuits].

APPENDIX B**In the Specification**

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03410 filed September 29, 2000, which in turn claims priority of German Patent Application Nos. 199 47 910.0 filed October 6, 1999 and 199 49 726.5 filed October 15, 1999.

In the Abstract

Please amend the abstract to read as follows:

The invention relates to a method for producing cellulosed shaped bodies. (a) Cellulose and an aqueous aminoxide are mixed, whereby a suspension is produced. (b) A cellulose solution is produced from the suspension, whereby water evaporates. (c) A first property and a second property of the produced cellulose solution is measured and the measured value is used for correcting the composition of the solution and (d) the cellulose solution is extruded through a gap of air and into an aqueous regenerating bath where said solution is coagulated to form shaped bodies.

In the Claims

Please amend claims 1-10 to read as follows:

1. A process for controlling the composition of a solution in the production of cellulosic shaped bodies, the process comprising:
 - (a) mixing cellulose and a water-containing amine oxide to form a suspension,
 - (b) creating a cellulose solution from the suspension by vaporization of water,
 - (c) measuring a first property of the cellulose solution and a second property different from the first property wherein the measured values of the first and second properties are used to calculate the actual composition of the solution and their deviation from values of a target composition is used to adjust the composition of the solution; and
 - (d) extruding the cellulose solution through an air gap into an aqueous regeneration bath where it is coagulated to form shaped bodies that are capable of undergoing further processing steps.
2. The process according to claim 1, wherein the first and second properties of the cellulose solution are selected from the group consisting of: relative permittivity, inductive conductivity, microwave absorption, refractive index, density, water content, and ultrasonic speed.
3. The process according to claim 2, further comprising measuring the cellulose solution's temperature, and the measured values of the first and second properties are adjusted on the basis of the measured temperature,

wherein the timing of the temperature measuring is selected from the group consisting of during the measuring of the first and second property, shortly before the measuring of the first and second property and after measuring of the first and second property.

4. The process according to claim 2, wherein at least one of the first and second property is measured in-line.
5. The process according to claim 2, wherein the solution composition is adjusted by manual intervention in the dosing of the suspension components of step (a).
6. The process according to claim 2, wherein operating conditions are adjusted by manual intervention in the operating conditions of step (b).
7. A device for performing a process for controlling the composition of a cellulose solution comprising cellulose and a water-containing amine oxide used in the production of cellulosic shaped bodies the device comprising:

(a) a slurring apparatus communicatively connected with at least two metering devices for metering input of the cellulose and water-containing amine oxide;

(b) a dissolving and vaporization arrangement connected to the slurring apparatus;

(c) an extruding device connected by a conduit to the dissolving and vaporizing arrangement and having a regeneration bath attached downstream therefrom;

(d) at least two measuring instruments contacting the conduit for measuring a first and a second property of the cellulose solution passing through the conduit; and

- (e) a controlling circuit for controlling the composition of the cellulose solution by determining the actual composition of the cellulose solution in the conduit from the measured values of the first and second property and to determine the deviation from a preset target composition and adjust metering devices to correct for any deviation.
8. The device according to claim 7, where in the first and second property of the cellulose solution are different and selected from the group consisting of: relative permittivity, inductive conductivity, microwave absorption, refractive index, density, water content, and ultrasonic speed.
 9. The device according to claim 8, further comprising a temperature measuring instrument for measuring the temperature of the cellulose solution and to compensate for temperature in the measured values of measuring instruments.
 10. The device according to claim 7, wherein the measuring instruments, a microprocessor and the metering devices form the controlling circuit.

METHOD AND DEVICE FOR PRODUCING CELLULOSED SHAPED BODIES

The invention relates to a process for controlling the composition of solutions in the production of cellulosic shaped bodies, in which (a) cellulose and a water-containing amine oxide are mixed to form a suspension, (b) a cellulose solution is created from the suspension by vaporization of water, (c) a first property or physical behavior of the cellulose solution formed thereby is measured and the measured value is used to adjust the composition of the solution, and (d) the cellulose solution is extruded through an air gap into an aqueous regeneration bath where it is coagulated to form shaped bodies that are capable of undergoing further processing steps. The invention further relates to a device for carrying out this process with a slurring apparatus equipped with metering devices, a dissolving and vaporizing arrangement connected to the slurring apparatus and an extruding device connected to the dissolving and vaporizing arrangement by a conduit and having a regeneration bath attached downstream. Wherever the text of this application refers generally to a property for measurement of the cellulose solution, this should also be understood to include a physical behavior of the solution that is capable of being measured (measured variable).

The properties of the cellulosic shaped bodies produced according to the amine oxide process, particularly the physical properties as textiles of the spun fibers and filaments, depend to a great extent on the composition of the spinning solution. It is therefore desirable for the achievement of optimal properties that the composition of

the spinning solution be monitored and that variations be kept within narrow tolerances. From WO 94/28212, it is known to take a sample from the spinning solution upstream from the extruding device and to measure its refractive index. In order to assure satisfactory extrusion products, the refractive index should be between 1.4890 and 1.491 at 60 °C. In addition to this discontinuous system of monitoring, a device for continuous measurement in the conduit, e.g. using a process refractometer, is also indicated. If the spinning solution presents a refractive index outside the refractive index cited, the feedstock for the premixer and/or the vaporization conditions must be reset.

The spinning solution is a ternary mixture of cellulose, amine oxide and water; therefore, its composition cannot be determined precisely by the measurement of only one property of the spinning solution. A number of solution compositions that fall within the solution area in the ternary system's triangle plot can be allocated to one refractive index. It is therefore not possible to control the concentration of the spinning solution precisely on the basis of the refractive index alone. The fact that a specific solution compound does not fall within the indicated refractive index range anyway is also not addressed.

The object of the present invention is to provide a process for manufacturing cellulosic shaped bodies in accordance with the amine oxide process, wherein the composition of the spinning solution can be monitored precisely. In particular, the composition of the spinning solution is to be monitored precisely so that even small

deviations from a predetermined target value can be used to correct the composition of the solution. In addition, a method for preparing cellulosic shaped bodies according to the amine oxide process is proposed, wherein the composition of the spinning solution is adjusted with the smallest possible delay or dead time, so that any fluctuations in the composition are eliminated rapidly or maintained within narrow limits. Finally, a device is also proposed for performing the process, by means of which changes in the spinning solution composition, e.g. due to changes in composition of the mixing materials, can be eliminated rapidly. Further advantages are explained in the following detailed description.

This object is solved by the process according to the invention indicated in the introduction in that, in step (c), a second property, which is different from the first property of the spinning solution, is measured, the actual composition of the solution is calculated on the basis of the two measured values, and the deviation of the actual composition from a target composition is used to adjust the composition of the solution.

Unlike the known process as described in WO 94/28212, two properties of the spinning solution are measured, thus allowing the composition to be calculated precisely from the measured values. This requires that the two properties of the pure components, cellulose, amine oxide and water are known in part. If they are not known, they must be determined experimentally, if necessary also by extrapolation. The measurement of the two properties according to the invention yields the exact composition of the solution. Accordingly, in the event of even small

deviations from a desired target composition in the solution, adjustments can be made to the dosages of the solution components for producing the solution, or to the operation forming the solution to compensate for the deviation. The process according to the invention thus allows of considerably more rigorous control of the solution composition than is possible if the refractive index alone is monitored and the adjustment is only activated after the value passes outside the predetermined tolerance range of the refractive index.

In the preferred embodiment of the process according to the invention, the two properties of the spinning solution are chosen from the relative permittivities, the electrical conductivity, the microwave intensity attenuation and speed, the refractive index, the density, the water content, and the ultrasonic speed. The refractive index and the density of the solution are particularly suitable for use as the two properties for measurement. The density can be measured either radiometrically or with a Coriolis force sensor. The water content can be determined by the Karl Fischer method.

In the preferred embodiment of the process according to the invention, the temperature of the cellulose solution is measured during or shortly before or after the two indicated properties are measured, and the measured values of the two solution properties or the physical behavior are adjusted and the measured values are adjusted on the basis of the measured temperature. Since the temperature of the spinning solution may vary, the measured values must be converted to the reference

temperature for which the property values or behavior values of the spinning solution of the target composition are known, with which the measured values are to be compared. This reference temperature, to which the measured values must be converted, is, for example 50 °C or 60 °C.

At least one of the two properties of the spinning solution is preferably measured in-line. In-line measurements not only allow rapid calculation of the solution composition, but they also limit the safety hazard from the possible initiation of uncontrollable runaway reactions due to the tendency of the solution to decompose.

In one embodiment of the process according to the invention, the solution composition is adjusted in the dosing of the components described in step (a) above. For example, if a variation is noted in the cellulose concentration of the spinning solution, it is necessary to make an alteration in the dosing system. The causes may be attributed to a change in the water content of the cellulose used, or in the solvent mixture N-methylmorpholine-N-oxide (NMMO)/ H₂O.

In a further embodiment, the adjustment to the solution composition is made to the operating conditions in step (b). If measurements reveal a shift in the NMMO/ H₂O ratio, this can be corrected in step (b), for example, by increasing or reducing the rate of water vaporization at this point.

Moreover, the device according to the invention described

in the introduction also presents a solution to the object of the invention in that a measuring apparatus is disposed in the conduit between the dissolving and vaporizing arrangement and the extruding device for the purpose of measuring two different properties of the solution and constitutes part of at least one controlling circuit for controlling the composition of the spinning solution. The measuring apparatus in the conduit between solution production and solution extrusion transmits signals, such as for example the refractive index and the density of the solution, by means of which a change is effected in the correcting variables, e.g. the dosing rates for cellulose and/or solvent, or the rate of water vaporization in the dissolving arrangement. The measuring apparatus consists of two instruments for measuring the different solution properties. The measuring instruments may be connected in series or in parallel. They are disposed in the conduit and provide results of measurement with no time so that dead time in the controlling circuit, and thus also fluctuations in the solution composition, are minimized. The invention also extends to the o-line arrangement of the measurement apparatus.

The measurement apparatus preferably includes a device for measuring the temperature of the solution and for compensating for the measured values of the indicated measuring instruments. In this way, the measurement apparatus returns measured values for both properties that have already been adjusted to compensate for temperature, so that these values can be compared with the property values of the spinning solution having the target composition with respect to a predetermined

temperature.

The purposes of the invention are served if the measurement apparatus, together with a microprocessor and the metering devices or the dissolving and vaporizing arrangement, together form at least one control circuit. In such event, correcting variables may be, for example, the dosing rates of the metering devices or the rate of water vaporization in the dissolving arrangement.

In the following, the invention will be explained in detail with reference to the drawing and a number of experiments.

The only figure shows the schematic flow diagram of an embodiment of the device according to the invention.

Water-containing cellulose and an NMMO/H₂O mixture are introduced into a continuous slurring apparatus 2 through a metering system 1. Apparatus 2 may be a multishaft apparatus as described in DE-C 198 37 210.8. A dissolving arrangement 3 is connected downstream of apparatus 2, wherein the suspension formed in 2 is transformed into a solution by the application of heat and negative pressure to vaporize the water. A suitable process for this purpose is known from DE-A 44 41 468.8. An extrusion tool 4 is connected to dissolving arrangement 3 by means of a conduit 8. The spinning solution is extruded by the extruding tool 4 across an air gap and into a regeneration bath 5. Depending on the type and design of the extrusion tool 4 and the subsequent operations, films, fibers, filaments or other shaped bodies may be produced. The shaped body 6 obtained

thereby is passed through a discharging device 7 to subsequent treatment steps such as washing out the amine oxide, secondary treatment, cutting, drying.

The invention provides for the disposition of a measuring apparatus 9-11 in conduit 8 between the dissolving arrangement 3 and the extrusion tool 4. This consists of a measuring instrument 9 for measuring a first property of the solution, a measuring instrument 10 for measuring a second property different from the first property, and an instrument 11 for measuring the temperature of the spinning solution in conduit 8 between measuring instruments 9 and 10. As is shown, measuring instruments 9 and 10 are disposed consecutively in the flow. Measuring instruments 9 and 10 transmit signals to microprocessor 12 that are already corrected on the basis of the temperature measurements provided by instrument 11. From the signals it receives from measuring instruments 9 and 10, microprocessor 12 calculates the actual composition of the solution in conduit 8, derives the deviation from the preset target composition of the spinning solution and, from this deviation, calculates the correcting variables for metering devices 1^a, 1^b and/or the operating conditions for dissolving arrangement 3. The correcting commands from microprocessor 12 are transmitted across one of signal circuits 13, 14, 15 or other signal circuits to the adjusting mechanisms concerned. Thus is demonstrated that, if the solution composition in conduit 8 deviates from the target value, the control mechanism, particularly the direct temperature compensation, according to the invention of the two measured values, allows for rapid correction, particularly since a sample

does not have to be withdrawn in order for the temperature adjustment to be made.

Experiment 1

A measurement apparatus consisting of an RIS 60-3 radiation process refractometer manufactured by Risens GmbH, Jena, and an LB 386 radiometric densitometer manufactured by EG & G Berthold, Bad Wildbad, were built into the spinning solution conduit directly downstream of the dissolving apparatus in a continuously running installation for spinning Lyocell batch fibers corresponding to the Figure. Measurements of the refractive index and the density were adjusted to reflect a temperature of 50 °C in each case. From the measured values thus obtained, the percentage composition of the spinning solution was calculated. Directly after the measurement apparatus, a sample was withdrawn from the conduit through a nozzle for removing samples and tested for cellulose content (precipitation method) and water content (Karl Fischer titration). The calculated values for the composition obtained therefrom and the values for cellulose and water determined by analysis are shown in the table. A good correspondence is revealed between the composition as calculated from the two measured properties, and the composition determined by analysis.

Experiment 2

The starting conditions were the same as for experiment 1. However, the vaporization conditions were then modified in dissolving arrangement 3 such that the water was vaporized more vigorously. To this end, the vacuum in

the dissolving arrangement was increased while the temperature profile and the ratio cellulose/NMMO/H₂O in the slurry were kept constant. The measured values for the properties obtained from the spinning solution, the values for cellulose, NMMO and water content derived therefrom, and the water and cellulose content values determined by analysis are shown in the table. This shows that the change to the vaporization conditions is reflected not only in the values obtained by direct analysis, but also in the data calculated from the solution properties.

Experiment 3

Beginning with the conditions established for experiment 2, and under constant conditions in the dissolving arrangement, the parameters for slurry preparation were temporarily changed such that the proportion of cellulose in the cellulose/NMMO/H₂O ratio was increased. The ratios that existed at the outlet from the dissolving arrangement after 35, 45 and 50 minutes were determined by direct analysis and by the percentage content as calculated on the basis of the properties measurements from the measurement apparatus as in experiment 1. These results are listed in rows 3a, 3b, 3c in the table. From the data presented, it is clear that temporary changes in the composition of the spinning solution can be calculated very precisely on the basis of measurements of the two solution properties.

The results of experimentation indicate that the solution composition can be calculated with extremely high accuracy from the measured values of two properties of

the spinning solution, so that close control of the composition of the spinning solution is possible. Since the calculated values are available with practically no delay, the process enables finer tolerances in the composition and correspondingly reduced fluctuations in the physical properties of the shaped body formed therefrom.

Exp. no.	Values measured by the measurement apparatus, temp.-adjusted to 50 °C		Values calculated from the measured values %/mass			Values determined by analysis %/mass	
	Refracti ve index	Densit y g/ml	Cellulo se	NMMO	Wate r	Cellulo se	Water
1	1.4847	1.2073	12.00	73.1 1	14.8 9	11.95	15.02
2	1.4885	1.2127	12.30	74.9 0	12.8 0	12.32	12.75
3a	1.4890	1.2144	12.69	74.5 6	12.7 5	12.70	12.72
3b	1.4895	1.2162	13.13	74.1 8	12.6 9	13.17	12.70
3c	1.4897	1.2168	13.28	74.0 5	12.6 7	13.25	12.68

Claims

1. A process for controlling the composition of a solution or solutions in the production of cellulosic shaped bodies, in which

(a) cellulose and a water-containing amine oxide are mixed to form a suspension,

(b) a cellulose solution is created from the suspension by vaporization of water,

(c) a first property or physical behavior of the cellulose solution formed thereby is measured and the measured value is used to adjust the composition of the solution, and

(d) the cellulose solution is extruded through an air gap into an aqueous regeneration bath where it is coagulated to form shaped bodies that are capable of undergoing further processing steps,

characterized in that

in step (c), a second property of the cellulose solution, which is different from the first property, is measured,

the actual composition of the solution is calculated on the basis of measured values for the two properties, and their deviation from a target composition is used to adjust the composition of the solution.

2. A process according to claim 1, characterized in

that the two properties of the spinning solution are chosen from the relative permittivities, the inductive conductivity, the microwave absorption, the refractive index, the density, the water content, and the ultrasonic speed.

3. A process according to claim 1 or 2, characterized in that the temperature of the cellulose solution is measured during or shortly before or after the two properties are measured, and the measured values of the two properties are adjusted on the basis of the measured temperature.

4. A process according to any of claims 1 to 3, characterized in that at least one of the two properties is measured in-line.

5. A process according to any of claims 1 to 4, characterized in that the solution composition is adjusted by manual intervention in the dosing of the suspension components of step (a).

6. A process according to any of claims 1 to 5, characterized in that the operating conditions are adjusted by manual intervention in the operating conditions of step (b).

7. A device for performing the process according to any of claims 1 to 6, having

a slurring apparatus (2) furnished with metering devices (1^a, 1^b),

a dissolving and vaporization arrangement (3) connected to the slurring apparatus (2) and

an extruding device (4) connected by a conduit (8) to the dissolving and vaporizing arrangement (3) and having a regeneration bath (5) attached downstream,

characterized in that a measuring apparatus (9-11) that constitutes part of at least one controlling circuit for controlling the composition of the spinning solution is disposed in the conduit (8) for the purpose of measuring two different properties of the cellulose solution.

8. A device according to claim 7, characterized in that the measuring apparatus consists of two instruments (9, 10) for the purpose of measuring different properties of the solution.

9. A device according to claim 8, characterized in that the measuring apparatus includes an instrument (11) for the purpose of measuring the temperature of the solution and compensating for temperature in the measured values of instruments (9, 10).

10. A device according to any of claims 7 to 9, characterized in that the measurement apparatus (9-11) together with a microprocessor (12) and the metering devices (1^a, 1^b) or the dissolving and vaporizing arrangement (3) forms control circuits.

ABSTRACT

The invention relates to a method for producing cellulosed shaped bodies. (a) Cellulose and an aqueous aminoxide are mixed, whereby a suspension is produced. (b) A cellulose solution is produced from the suspension, whereby water evaporates. (c) A first property and a second property of the produced cellulose solution is measured and the measured value is used for correcting the composition of the solution and (d) the cellulose solution is extruded through a gap of air and into an aqueous regenerating bath where said solution is coagulated to form shaped bodies.

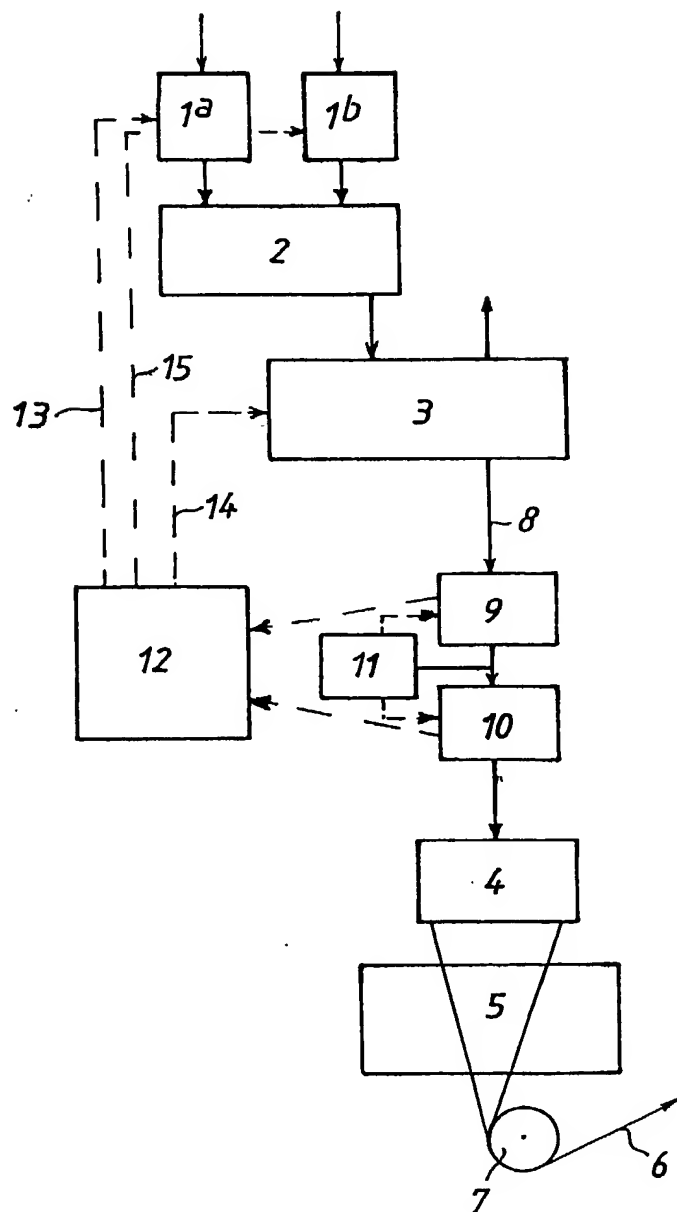


Figure 1

PATENT APPLICATION

ALC 6/us

DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATION

ATTORNEY DOCKET NO. 4197-112

As a below named inventor, I hereby declare that:

My residence/post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD AND DEVICE FOR PRODUCING CELLULOSED SHAPED BODIES

the specification of which is attached hereto unless the following box is checked:

(X) was filed March 4, 2002 as US Application Serial No. 10/070,624 or PCT International Application
Number _____ and was amended on _____ (if applicable).

I hereby state that I have reviewed and understood the contents of the above-identified specification, including the claims, as amended by any amendment(s) referred to above. I acknowledge the duty to disclose all information which is material to patentability as defined in 37 CFR 1.56.

Foreign Application(s) and/or Claim of Foreign Priority

I hereby claim foreign priority benefits under Title 35, United States Code Section 119(a-d) or 365(b) of any foreign application(s) for patent or inventor(s) certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor(s) certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NUMBER	DATE FILED	PRIORITY CLAIMED UNDER 35 U.S.C. 119
Germany	199 47 910 0	6 October 1999	YES: <input checked="" type="checkbox"/> NO: <input type="checkbox"/>
Germany	199 49 726 5	12 October 1999	YES: <input checked="" type="checkbox"/> NO: <input type="checkbox"/>
PCT	PCT/DE00/03410	29 September 2000	YES: <input checked="" type="checkbox"/> NO: <input type="checkbox"/>

U.S. Priority Claim

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NUMBER	FILING DATE	STATUS(patented/pending/abandoned)

POWER OF ATTORNEY.

I, as a named inventor, I hereby appoint the following attorney(s) and/or agent(s) listed below to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

Steven J. Hultquist, Reg. No. 28021

Marlaine Fuierer, Reg. No. 39983

Send Correspondence to: Steven J. Hultquist Intellectual Property/Technology Law P.O. Box 13325 Research Triangle Park, NC 27709	Direct Telephone Calls To: Steven J. Hultquist (919) 419-9350
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1-00
I Name of First Inventor: Frank Gunter NiemezResidence: An der Brücke 19, D-07407 Rudolstadt, GermanyCitizenship: GermanPost Office Address: SameInventor's Signature: Frank Gunter NiemezDate: 25.04.2002

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DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATION (continued)

ATTORNEY DOCKET NO. 4137-112

2.00
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Date

28.05.02